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Indian Standard

SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(*Third Revision*)

Second Reprint SEPTEMBER 2007
(Including Amendment Nos. 1, 2, 3 & 4)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Gr 5

April 1986

AMENDMENT NO. 4 MAY 2002
TO
IS 1462 : 1985 SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(Second Revision)

[*Page* 19, *clauses* **B-4** and **B-5**] — Substitute the following for the existing:

B-4 NUMBER OF TESTS

B-4.1 Tests for all the requirements given in 2 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to this specification if the composite sample satisfies all the requirements given in 2.

(PCD 19)

Printed at Shri Ganesh Associates, Delhi

AMENDMENT NO. 3 APRIL 1998
TO
IS 1462 : 1985 SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(Third Revision)

(*Page* 9, *clause* A-7) — Substitute the following for the existing matter:

A-7 DETERMINATION OF MAGNESIUM

A-7.1 Principle — On fusion with anhydrous sodium carbonate, magnesium silicate gets converted into magnesium oxide which is then titrated against Standard EDTA solution.

A-7.2 Reagents

A-7.2.1 *Concentrated Hydrochloric Acid*

A-7.2.2 *Strong Ammonia Solution*

A-7.2.3 *Anhydrous Sodium Carbonate*

A-7.2.4 *Standard EDTA Solution* — 0.05 M.

A-7.2.5 *Strong Ammonia* — Dissolve 6.75 g of ammonium chloride in 74 ml of strong Ammonia solution and dilute to 100 ml with water.

A-7.3 Procedure

Weigh accurately a clean, dry platinum crucible containing approximately about 0.2 g of anhydrous sodium carbonate. Weigh in accurately about 0.12 g of the Talc sample, followed by a further quantity of approximate 0.4 g of anhydrous sodium carbonate. Fuse the mixture by heating the crucible with the lid over a flame for about 30 minutes. Extract the fused mass with instalments of about 3-4 ml of concentrate. Hydrochloric acid (crush the fused mass with a glass rod to facilitate the extraction) and transfer quantitatively to 150-ml beaker. Wash the crucible and the lid with water and transfer to the beaker. Dissolve by stirring and gentle crushing with the glass rod and boil, if required for complete digestion. Cool and neutralise by slowly adding ammonia solution till a white precipitate appears. Add a little excess of ammonia. Cool and filter through a wet filter paper into 500-ml conical flask. Give washings with distilled water. Add 10-12 ml strong Ammonia — Ammonium Chloride solution, a pinch of solochrome black indicator and titrate against standard 0.05 M EDTA solution to a blue end point.

A-7.4 Calculation

$$\text{Magnesium as MgO, percent by mass} = \frac{V \times 0.002\,015 \times M \times 100}{W \times 0.05}$$

where

V = volume of 0.05 M EDTA solution,

M = molarity of EDTA solution, and

W = weight of talc taken for fusion in g.

(PCD 19)

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AMENDMENT NO. 2 MAY 1993
TO
IS 1462 : 1985 SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(*Third Revision*)

[*Page* 5, *Table* 1, *Sl No.* (vi), *col* 2] — Substitute 'Magnesium (as MgO), percent by mass, *Min*' *for* 'Magnesium (as MgO), percent by mass, *Max*'.

(PCD 19)

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AMENDMENT NO. 1 MAY 1992
TO
IS 1462 : 1985 SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(Third Revision)

[*Page* 5, *Table* 1, *Sl No.* (xii)] — Insert the following new requirement at the end:

(1)	(2)	(3)	(4)
'xiii)	Loss on drying, percent by mass, *Max*	1	A-14'

(*Page* 16, *clause* **A-13.2**) — Insert the following new clause after **A-13.2**:

'A-14 DETERMINATION OF LOSS ON DRYING

A-14.1 Procedure

Weigh accurately about 5 g of the material in a weighed, clean and dry squat form weighing bottle and dry to constant mass at 105 ± 2°C in an oven.

A-14.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100\,m}{M}$$

where

m = loss in mass, in g, on drying, and

M = mass, in g, of the material taken for the test.'

(PCD 19)

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Indian Standard

SPECIFICATION FOR TALC FOR COSMETIC INDUSTRY

(Third Revision)

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(*Continued on page* 20)

Indian Standard

SPECIFICATION FOR
TALC FOR COSMETIC INDUSTRY

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Indian Standards Institution on 11 November 1985, after the draft finalized by Cosmetics Sectional Committee had been approved by the Petrolum, Coal and Related Products Division Council.

0.2 This standard was originally published in 1959 and subsequently revised in 1967 and 1977. Initially while' reviewing this standard the Sectional Committee had agreed to indicate separately the essential and optional requirements. Subsequently this was found non-implementable for certification and therefore, in this revision, only a single set of requirement has been stipulated.

0.3 Based on interlaboratory testings, the limits of acid soluble iron has been revised and a new requirement of acid soluble matter has been introduced in this revision.

0.4 In cosmetics prepared from talc, freedom from grit is an important attribute. In the absence of any reproducible method of test for this characteristic, the following method may be used:

> Take a 20 g sample of the material in a beaker and wet it with a little rectified spirit. Remove by over-flow in a carefully controlled steady stream of water, a large portion of the material. The grit being heavier will remain in the beaker along with some talc. Test the residue by rubbing between the finger and the thumb for the presence of grit.

0.5 In the preparation of this standard, assistance has been taken from specification No. 10 'Talc', issued by the Toilet Goods Association, USA, 1971.

0.6 In the cosmetic industry, talc is used in the manufacture of talcum powder, face powder, depilatories and cold and other creams.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 The standard prescribes the requirements and methods of sampling and test for talc used in cosmetic industry.

2. REQUIREMENTS

2.1 Description — The material shall be finely powdered mineral consisting essentially of hydrated magnesium silicate. It shall be odourless and shall be free from gritty particles (*see* **0.3**).

2.1.1 The whiteness, slip and lustre of the material shall be as agreed to between the purchaser and the supplier.

2.1.1.1 The colour of the material shall not change when heated for 5 minutes at 200°C.

2.2 The material shall also comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the Table 1.

3. PACKING AND MARKING

3.1 The material shall be packed in well-closed containers as agreed to between the purchaser and the supplier.

3.2 The packages shall be securely closed and legibly marked with the following information:

a) Name of the material;
b) Manufacturer's or supplier's name and recognized trade-mark, if any;
c) Mass of the material in the package; and
d) Batch number to enable the lot of manufacture to be traced back from records.

---

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR TALC FOR COSMETIC INDUSTRY

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Fineness:		
	a) Retained on 150-micron IS sieve, percent by mass, *Max*	0·1	A-2
	b) Retained on 90-micron IS sieve, percent by mass, *Max*	2·0	
	c) Analysis on sieves finer than 90 microns	To be reported	
ii)	*p*H of aqueous suspension, *Max*	9·5	A-3
iii)	Matter soluble in water, percent by mass, *Max*	0·2	A-4
iv)	Heavy metals (as Pb), ppm, *Max*	20	A-5
v)	Arsenic (as As_2O_3), ppm, *Max*	2	A-6
vi)	Magnesium (as MgO), percent by mass, *Max*	20·0	A-7
vii)	Bulk density	As agreed to between the purchaser and the supplier	A-8
viii)	Loss on ignition, percent by mass, *Max*	7·0	A-9
ix)	Carbonates	To pass the test	A-10
x)	Acid soluble iron, percent by mass, *Max*	0·75	A-11
xi)	Water soluble iron salts	To pass the test	A-12
xii)	Acid soluble matter, percent by mass, *Max*	6	A-13

3.2.1 The product may also be marked with Standard Mark.
Mark.

3.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of preparing representive test samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHODS OF TEST FOR TALC FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF FINENESS

A-2.1 Procedure for Material Retained on 150-Micron IS Sieve — Place about 10 g of the material, accurately weighed, in 150-micron IS sieve and wash by means of slow stream of running tap water and finally with fine stream from a wash bottle until all the material that can pass through the sieve has passed. Let the water drain from the sieve and then dry the sieve containing the residue on a steam bath. Carefully transfer the residue, on to a tared watch glass, dry it to constant mass at 105 ± 2°C and weigh.

A-2.1.1 *Calculation*

$$\text{Material retained on the specified sieve, percent by mass} = 100 \frac{m}{M}$$

where

m = mass in g of the residue retained on the sieve, and
M = mass in g of the material taken for the test

A-2.2 Procedure for Material Retained on 90-Micron IS Sieve — Carry out the test as prescribed under **A-2.1**, using a fresh quantity of the material and 90-micron IS sieve. Calculate in the manner given under **A-2.1.1**.

---

*Specification for water for general laboratory use (*second revision*).

A-2.3 Procedure for Analysis on Sieves Finer Than 90-Microns — Carry out the procedure prescribed in **A-2.1**, using a fresh quantity of the material and IS sieves as agreed to between the purchaser and the supplier.

A-3. DETERMINATION OF *p*H

A-3.1 Outline of the Method — An aqueous extract of the material is tested for determining the *p*H value.

A-3.1 Apparatus

A-3.1.1 *p*H *Meter* — equipped with glass electrode.

A-3.2 Procedure — Boil about 10 g of the material, accurately weighed, with 50 ml of water for 30 minutes, adding water from time to time to maintain approximately the original volume of the liquid. Cool and filter. Determine the *p*H of the filtrate within 5 minutes of filtration and preserve the filtrate for test under **A-4**.

A-4. DETERMINATION OF MATTER SOLUBLE IN WATER

A-4.1 Procedure — Evaporate the filtrate preserved under **A-3.2** in a tared evaporating dish on a steam-bath and finally dry constant mass in an oven at 105 ± 2°C.

A-4.2 Calculation

$$\text{Matter soluble in water, percent by mass} = 100\,\frac{m}{M}$$

where

m = mass in g of the residue obtained, and
M = mass in g of the material taken for the test under **A-3.2**.

A-5. TEST FOR HEAVY METALS

A-5.0 Outline of the Method — The brown colour produced by the material with hydrogen sulphide solution is matched against that produced with standard lead solution.

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders* — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *Perchloric Acid* — 60 percent (*m/m*).

A-5.2.2 *Hydrofluoric Acid* — 40 percent (*m/m*).

A-5.2.3 *Citric Acid* — Solid.

A-5.2.4 *Ammonium Hydroxide* — Dilute one volume of liquor ammonia (relative density 0·90) with 10 volumes of water.

A-5.2.5 *Hydrogen Sulphide Solution* — Saturated solution, freshly prepared.

A-5.2.6 *Standard Lead Solution* — Dissolve 1·600 g of lead nitrate in water and make up the solution to exactly 1 000 ml. Pipette out 10 ml of the solution and dilute it again with water to 1 000 ml. One millilitre of the final solution contains 0·01 mg of lead (Pb). The solution should be freshly prepared.

A-5.3 Procedure — Place 2·000 g of the material, accurately weighed, in a platinum dish and incinerate for about 2 hours at 525 to 550°C. Cool and treat with 5 ml of perchloric acid. Take to fumes on a hot-plate with three successive portions of hydrofluric acid. Cool and dilute with water. Filter the solution, if necessary, with suction, through a fine fritted glass filter catching the filtrate in a 500-ml glass-stoppered conical flask. Transfer the solution to a Nessler cylinder. Add 10 g of citric acid and adjust to *p*H 3·0 to 3·4 by ladding dilute ammonium hydroxide (yellow to purple colour with bromophenol blue indicator). Add 10 ml of hydrogen sulphide solution and mix. Carry out simultaneously a control test using 4 ml of standard lead solution and the same quantities of other reagents as used with the material. Allow the cylinders to stand for 10 minutes and compare the colour in the two cylinders.

A-5.3.1 The material shall be taken as having satisfied the requirement prescribed in Table 1 if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-6. TEST FOR ARSENIC

A-6.0 Outline of the Method — The stain obtained on a mercuric bromide paper by arsenic is compared with that obtained with standard arsenic solution.

A-6.1 Reagents

A-6.1.1 *Concentrated Sulphuric Acid* — analytical reagent grade (*see* IS : 266-1977*).

A-6.1.2 *Concentrated Nitric Acid* — analytical reagent grade (*see* IS : 264-1976†).

---

*Specification for sulphuric acid (*second revision*).

†Specification for nitric acid (*second revision*).

A-6.1.3 *Hydrofluoric Acid*

A-6.1.4 *Mixed Acid* — Dilute one volume of concentrated sulphuric acid with four volumes of water, and to this add 10 g of sodium chloride for each 100 ml of the solution.

A-6.1.5 *Ferric Ammonium Sulphate Solution* — Dissolve 84 g of ferric ammonium sulphate in water with 10 ml of mixed acid and make up to 1 litre.

A-6.1.6 *Concentrated Hydrochloric Acid* — analytical reagent grade (*see* IS : 265-1976*).

A-6.1.7 *Stannous Chloride Solution* — Dissolve 80 g of stannous chloride in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

A-6.2 Procedure

A-6.2.1 Place 1·000 g of the material, accurately weighed, in a platinum dish and incinerate for about 2 hours at 525 to 550°C. Cool and treat with a mixture of 5 ml concentrated sulphuric acid and 5 ml of concentrated nitric acid. Take to fumes on hot-plate. Cool and again take so fumes with three succesive portions of hydrofluoric acid. Cool and dissolve cautiously in water and make up the volume to exactly 100 ml.

6.2.2 Carry out the test for arsenic as directed in IS : 2088-1983†, taking 10 ml of the solution prepared in **A-6.2.1** into a Gutzeit bottle, then adding to it 20 ml of water, 2 ml of ferric ammonium sulphate solution and 0·5 ml of stannous chloride solution. Compare the stain obtained with that produced with 0·000 2 mg of arsenic trioxide (As_2O_3).

A-7. DETERMINATION OF MAGNESIUM

A-7.1 Outline of the Method — After precipitation of silica, magnesium is precipitated as magnesium ammonium phosphate which is ignited and weighed as the pyrophosphate.

A-7.1 Reagents

A-7.1.1 *Anhydrous Sodium Carbonate*

A-7.1.2 *Concentrated Hydrochloric Acid* — (*See* IS : 265-1976‡).

A-7.1.3 *Dilute Hydrochloric Acid* — 1:3, 1:10 and 1:20 (*v/v*).

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*Specification for hydrochloric acid (*second revision*).

†Methods for the determination of arsenic (*second revision*).

‡Specification for hydrochloric acid (*second revision*).

A-7.1.4 *Ammonium Chloride*

A-7.1.5 *Bromine*

A-7.1.6 *Methyl Red Indicator* — Dissolve 0·1 g of methyl red in 100 ml of rectified spirit.

A-7.1.7 *Dilute Ammonium Hydroxide* — 1:1, 1:3, 1:10 and 1:19 (*v/v*).

A-7.1.8 *Ammonium Chloride Solution* — 2 percent.

A-7.1.9 *Ammonium Oxalate Solutions* — 4 percent and 0·1 percent (*m v*).

A-7.1.10 *Ammonium Phosphate*

A-7.1.11 *Concentrated Ammonium Hydroxide* — relative density 0·92.

A-7.3 Procedure

A-7.2-1 Transfer about 1 g of the material, accurately weighed, to a platinum crucible and fuse with 5 g of anhydrous sodium carbonate. Start the fusion over a small flame, then increase the heat gradually until the entire contents of the crucible are in a molten state and continue heating for 20 minutes. Keep a close fitting platinum lid on the crucible during the fusion. When fusion is complete, allow the crucible and contents to cool. During the cooling period, partially immerse the crucible several times in cold water to chill the outer portions of the melt as this facilities the subsequent removal of the melt. Do not allow the water to enter the crucible while the contents are hot because of possible spattering. Transfer the crucible to a 1 000 ml porcelain crucible containing 200 ml of water. Remove the crucible and lid taking care to scrub and rinse out any adhering particles of the melt. Carefully, acidify the contents of the crucible with concentrated hydrochloric acid, introducing the acid in small portions and keeping a watch-glass over the crucible to avoid loss by spattering. Add 30 ml excess of concentrated hydrochloric acid and evaporate to dryness on a steam-bath, breaking up any crust which may form. When the material appears to be completely dry and no odour of hydrochloric acid can be detected, remove the crucible from the steam-bath and allow to cool.

A-7.2.2 Wash down the sides of the crucible with 20 ml of concentrated hydrochloric acid and then with water. Repeat the evaporation as described in **A-7.2.1** and bake for one hour in an oven at 150°C. Cool the residue, drench with 25 ml of concentrated hydrochloric acid and 175 ml of water, and warm with stirring until all the soluble salts are dissolved. Filter off silica on a close texture filter paper, wash five times with dilute hydrochloric acid (1 : 20) and five times with hot water, collecting the combined filtrates in a 600-ml beaker.

A-7.2.3 Add 5 g of ammonium chloride and 3 ml of bromine to the filtrate and boil the solution to expel excess of bromine. Add 2 drops of methyl red indicator to the boiling solution followed by enough dilute ammonium hydroxide (1 : 1) to make it just alkaline, and boil for two minutes. Filter off the precipitate and collect the filtrate in a 600-ml beaker, wash the beaker and precipitate with ammonium chloride solution and add the washings to the filtrate. Reject the precipitate.

A-7.2.4 Slightly acidify the combined filtrates with concentrated hydrochloric acid and evaporate to about 250 ml. Add 1 ml of concentrated hydrochloric acid and precipitate the calcium by adding slowly to the boiling solution 50 ml of hot 4 percent ammonium oxalate solution, followed by dilute ammonium hydroxide (1:10) until it is neutral or slightly alkaline to methyl red. Test for complete precipitation by adding a little more of the oxalate solution. Allow the precipitate to settle for one hour. Filter off the precipitate and wash it three times with cold 0·1 percent ammonium oxalate solution. Set aside the filtrate.

A-7.2.5 Place the original beaker under the funnel, dissolve the precipitate in 25 to 50 ml of dilute hydrochloric acid (1 : 10) and wash the paper with hot water. Dilute the solution to 100 ml, if necessary, add 5 ml of 4 percent ammonium oxalate solution, boil and then add dilute ammonium hydroxide (1 : 1) dropwise, until the solution is just alkaline. Set aside for one hour, filter and combine with the filtrate set aside in **A-7.2.4**.

A-7.2.6 Adjust the volume of the combined filtrates from calcium precipitation (*see* **A-7.2.5**) to 400 ml, acidify slightly with dilute hydrochloric acid (1 : 3) and then add 10 g of ammonium phosphate. If the solution is alkaline, add dilute hydrochloric acid (1 : 3) to make the solution acidic. When the ammonium phosphate has completely dissolved, add dilute ammonium hydroxide (1 : 3) slowly while stirring vigorously with a glass rod till the solution turns yellow and a precipitate begins to appear. Avoid rubbing the sides of the beaker with the stirrer. Continue stirring and adding dilute ammonium hydroxide (1 : 3) until no further precipitation occurs. Add 50 ml of concentrated ammonium hydroxide and allow the solution to stand overnight.

A-7.2.7 Filter the solution through a filter paper and wash with dilute ammonium hydroxide (1 : 19). Carefully dissolve magnesium ammonium phosphate precipitate by alternately pouring through the filter dilute hydrochloric acid (1 : 3) and water. Collect the filtrate in a clean 400-ml beaker, adjust the volume to 250 ml and add 0·2 g of ammonium phosphate. Add slowly dilute ammonium hydroxide (1 : 3) to the filtrate while it is stirred rapidly. Continue stirring till precipitation is apparently complete. Add 25 ml of concentrated ammonium hydroxide and allow to stand overnight

A-7.2.8 Filter the solution through a close texture filter paper and wash ten times with dilute ammonium hydroxide (1 : 19). Transfer the precipitate wrapped in the moist paper to a weighed platinum crucible. Chart the precipitate slowly without allowing it to ignite and burn off the carbon over a gradually increased flame which should not heat the crucible to more than a faint red. Finally, heat at 1 000 to 1 100°C for 30 minutes. Cool the crucible in a desiccator and weigh. Repeat the procedure of heating, cooling and weighing until the difference between two successive weighings does not exceed 1 mg.

A-7.3 Calculation

$$\text{Magnesium oxide (as MgO), percent by mass} = \frac{m \times 0{\cdot}362\,1}{M} \times 100$$

where

m = mass in g of the residue, and
M = mass in g of the material taken for the test.

A-8. DETERMINATION OF BULK DENSITY

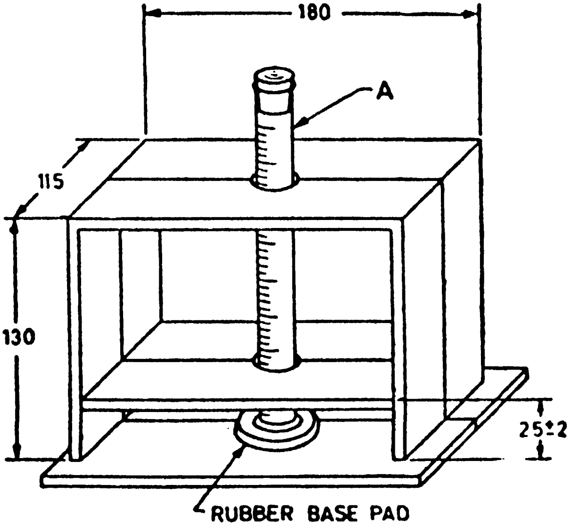
A-8.1 Apparatus

A-8.1.1 Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error, if any, of less than one millilitre. The distance between 0 and 250-ml graduation on the measuring cylinder shall not be less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the base of measuring cylinder and the rubber base pad, when the measuring cylinder is raised to the full height shall be 25 ± 2 mm.

A-8.1.2 The rubber base pad shall have a shore hardness of 42 to 50.

A-8.1.3 Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0·1 g.

A-8.2 Procedure — Sieve about 40 g of the material through 250-micron IS sieve on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder, and within one second lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper) and let it drop. Note the volume after dropping it once. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-8.3 Calculation

Bulk density in g/ml:

a) After one drop $= \frac{m}{V_1}$

b) After 50 drops $= \frac{m}{V_{50}}$

where

m = mass in g of the material taken for the test,
V_1 = volume in ml of the material after one drop, and
V_{50} = volume in ml of the material after 50 drops.

A-9. DETERMINATION OF LOSS ON IGNITION

A-9.1 Procedure — Weigh accurately about 4 g of the material in a tared crucible and ignite at red heat to constant mass. Cool in a desiccator and weigh.

A-9.2 Calculation

$$\text{Loss on ignition, percent by mass} = 100 \frac{(M - m)}{M}$$

where

m = mass in g of the ignited material, and
M = mass in g of the material taken for the test.

A-10. TEST FOR CARBONATES

A-10.0 Outline of the Method — The material is treated with dilute mineral acid and observed for any effervescence.

A-10.1 Reagents

A-10.1.1 *Dilute Hydrochloric Acid* — approximately 0·5 N.

A-10.2 Procedure — Mix about 5 g of the material, accurately weighed, with 50 ml of dilute hydrochloric acid in a 250-ml beaker. Cover the beaker with a watch-glass and warm the contents on a steam-bath. Observe if there is any effervescence, and proceed for test under **A-11**.

A-10.2.1 The material shall be taken as having passed the test if no effervescence takes place.

A-11. DETERMINATION OF ACID SOLUBLE IRON

A-11.0 Outline of the Method — All the iron is reduced to ferrous state and then titrated against potassium dichromate solution, using diphenylamine indicator.

A-11.1 Reagents

A-11.1.1 *Ammonium Hydroxide* — approximately 4 N

A-11.1.2 *Dilute Hydrochloric Acid* — approximately 4 N.

A-11.1.3 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-11.1.4 *Stannous Chloride Solution* — Dissolve 6 g of crystallized stannous chloride ($SnCl_2 . 2H_2O$) in 60 ml of concentrated hydrochloric acid and dilute with water to 100 ml. Keep this solution well stoppered.

A-11.1.5 *Mercuric Chloride Solution* — saturated.

A-11.1.6 *Titrating Mixture* — Mix 150 ml of concentrated sulphuric acid (*see* IS : 266-1977†) and 150 ml of phosphoric acid (relative density 1·75) and make up to 1 litre with water.

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*Specification for hydrochloric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

A-11.1.7 *Diphenylamine Indicator* — Dissolve 1 g of diphenylamine in 100 ml of contrated sulphuric acid.

A-11.1.8 *Standard Potassium Dichromate Solution* — 0·04 N (*see* IS : 2316-1968*).

A-11.2 Procedure

A-11.2.1 Digest the contents of the beaker (**A-10.2**) on a steam-bath for about half an hour and filter through a tared Gooch crucible. Wash the residue thoroughly with hot water. Reject the residue. Make the solution distinctly ammoniacal by the addition of ammonium hydroxide. Bring the solution to boil and allow to stand on steam-bath for half an hour. Filter through a filter paper and wash the precipitate thoroughly with hot water. Dissolve the precipitate by pouring hot dilute hydrochloric acid on the filter paper three or four times in small portions and collect the solution in a conical flask. Wash the filter paper thoroughly with water, collecting the washings also in the same conical flask.

A-11.2.2 Add about 10 ml of concentrated hydrochloric acid and heat the solution to boiling. Reduce the iron by adding stannous chloride solution drop by drop until the yellow colour of the solution just disappears. Do not add more than 2 to 3 drops of stannous chloride solution in excess. Allow the solution to cool and then add in one lot 10 ml of mercuric chloride solution and stir thoroughly. A white precipitate of mercurous chloride is obtained (if instead, a grey or black precipitate is obtained on addition of mercuric chloride, it indicates that reduction has not been effected satisfactorily and the test should be repeated). Dilute the solution to about 150 ml and add 15 ml of titrating mixture and 5 drops of diphenylamine indicator. Titrate with standard potassium dichromate solution until the colour of the solution changes to a deep bluish-violet which does not fade on stirring.

A-11.3 Calculation

$$\text{Iron (as } Fe_2O_3 \text{), percent by mass} = \frac{7{\cdot}985\, V N}{M}$$

where

V = volume in ml of standard potassium dichromate solution used in the titration,

N = normality of the potassium dichromate solution, and

M = mass in g of the material taken for the test in **A-10.2**.

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*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

A-12. WATER SOLUBLE IRON SALTS

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* — 100 ml capacity.

A-12.2 Reagents

A-12.2.1 *Citric Acid Solution* — 20 percent (*m/v*) solution of iron-free citric acid in water.

12.2.2 *Thioglycollic Acid*

A-12.2.3 *Ammonia Solution* — iron-free.

A-12.2.4 *Standard Iron Solution* — Take 0·173 g of ferric ammonium sulphate, add 1·5 ml of hydrochloric acid and dilute to 1 000 ml. One millilitre of this solution contains 0·02 mg of iron.

A-12.3 Procedure — Boil 5 g of the material with 15 ml of water for 30 minutes, make up any water lost by evaporation, and filter. The filtrate, after the addition of 5 ml nitric acid and dilution to 50 ml with water shall be tested as given below.

A-12.3.1 To the above solution add 2 ml of citric acid solution and 2 drops of thioglycollic acid, make alkaline with ammonia, dilute to mark with water, and allow to stand for 5 minutes. Carry out a control test in another Nessler cylinder using 2·0 ml of standard iron solution in place of the material and add 2 ml of citric acid solution, 2 drops of thioglycollic acid, make the solution alkaline with ammonia and dilute to mark with water. Compare the intensity of the colour produced in two cylinders.

A-12.3.2 The material shall be taken to have passed the test if the intensity of colour produced with the material is not greater than that obtained in the control test.

A-13. ACID SOLUBLE MATTER

A-13.1 Reagents

A-13.1.1 *Dilute Hydrochloric Acid* — approximately 4 N.

A-13.1.2. *Sulphuric Acid* — *See* IS : 266-1977*.

A-13.2 Procedure — Weigh 2 g of the material and digest with 40 ml of dilute hydrochloric acid for fifteen minutes. Filter, evaporate the filtrate, to the residue add 0·1 ml of sulphuric acid and ignite to constant weight.

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*Specification for sulphuric acid (*second revision*).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF TALC FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* 2.2)

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 50	3
51 " 150	4
151 " 300	5
301 " 500	7
501 and above	10

NOTE — When the number of containers in the lot is less than 3 or 3, the number of containers to be selected shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables (*see* IS : 4905-1968*) shall be used; in case such tables are not available, the following procedure shall be adopted:

> Starting from any container, count them as 1, 2, 3,......, etc. up to r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples — Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **2** and shall be not less than 0·2 kg.

B-3.1.1 Thoroghly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each of the selected container and shall be well mixed together so as to form a composite sample weighing not less than 0·5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place as agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

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*Methods for random sampling.

B-4. NUMBER OF TESTS

B-4.1 Test for magnesium (as MgO), percent by mass, shall be conducted on individual samples and for all the remaining characteristics given in Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The material shall be taken to have conformed to the requirements of magnesium (as MgO) if the expression $\bar{X} - 0{\cdot}6\, R$ is greater than or equal to the minimum value specified in Table 1. Where $\bar{X}$ is the average of the tests sesults and R is the difference between the maximum tand he minimum value of the tests results.

B-5.2 The material shall be considered as conforming to the requirements of remaining characteristics if none of the test conducted on composite sample fails.

(*Continued from page* 2)

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